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1996

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*Citation for published version (APA):*

Wessman, L. (1996). *Freezing and thawing of natural stone with different internal and external concentration of salt solution*. (Report TVBM (Intern 7000-rapport); Vol. 7102). Division of Building Materials, LTH, Lund University.

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1

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# **Freezing and Thawing of Natural Stone with Different Internal and External Concentration of Salt Solution**

**Lubica Wessman**

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**Report TVBM-7102**

**Lund, September 1996**



## Abstract

Sandstone and limestone have been frozen and thawed with different internal and external concentrations of solutions of NaCl and  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ . Tested concentrations were 0%, 0.25%, 0.5% and 1.0% by weight.

The external damage was measured as weight loss and the internal damage was measured as a loss in dynamic modulus of elasticity. Both external and internal damage increased linearly with number of freeze-thaw cycles. There is no correlation between internal and external damage.

External concentration had a considerable effect on weight loss - pure water gave almost no damage while external salt solution gave rise to considerable damage. No significant difference was noted for the different external salt concentrations. Internal salt concentration had almost no effect on weight loss.

Neither the internal nor the external concentration normally have effect on the internal damage.

## 1. Introduction

Ancient buildings and monuments made of natural stone deteriorate because of different processes influenced by nature and human action. Conservation and restoration of such cultural heritage is causing governments all over the world considerable cost, as it is considered important to preserve these relics of the past to future generations.

To make a proper conservation, or any other measure to preserve an object, it is important to have knowledge of the processes that affect deterioration. One such process is frost. Frost action is probably even more harmful together with salt [1].

Salt is often removed when a stone object is cleaned. Sometimes not only the efflorescence visible on the surface to the eye are removed, but effort is also made to remove the salt from the pore surface, e.g. by application of a damp clay covering, into which salts can migrate.

When it is raining on a stone object, the concentration of salts and other air pollutions in the pore water and in the surface water is not necessarily the equal. The aim of this work is to study how different internal and external salt concentrations affect the damage caused by freezing and thawing. Testing of concrete in NaCl-solution has shown that the concentration of the solution inside the material has smaller effect than the concentration of the solution surrounding the material [2]. This work is based on the hypothesis that this is valid also for natural stone.

The results presented here can hopefully contribute to better knowledge of the importance of removing salts from stone objects to be preserved.

## 2. Used stones and salts

### 2.1 Sandstone

The tested sandstone is a calcite bounded type from Gotland called Valar. It has a porosity of about 17%. Is light grey, normally with no tints in it, but sometimes it contains light brown lines parallel to the bedding. These lines contain larger amounts of clay minerals than does the light grey parts. Thin section microscopy of the stone shows that it consists almost entirely of quartz grains of the size 0.05-0.15 mm with empty spaces between them constituting the porosity [4].

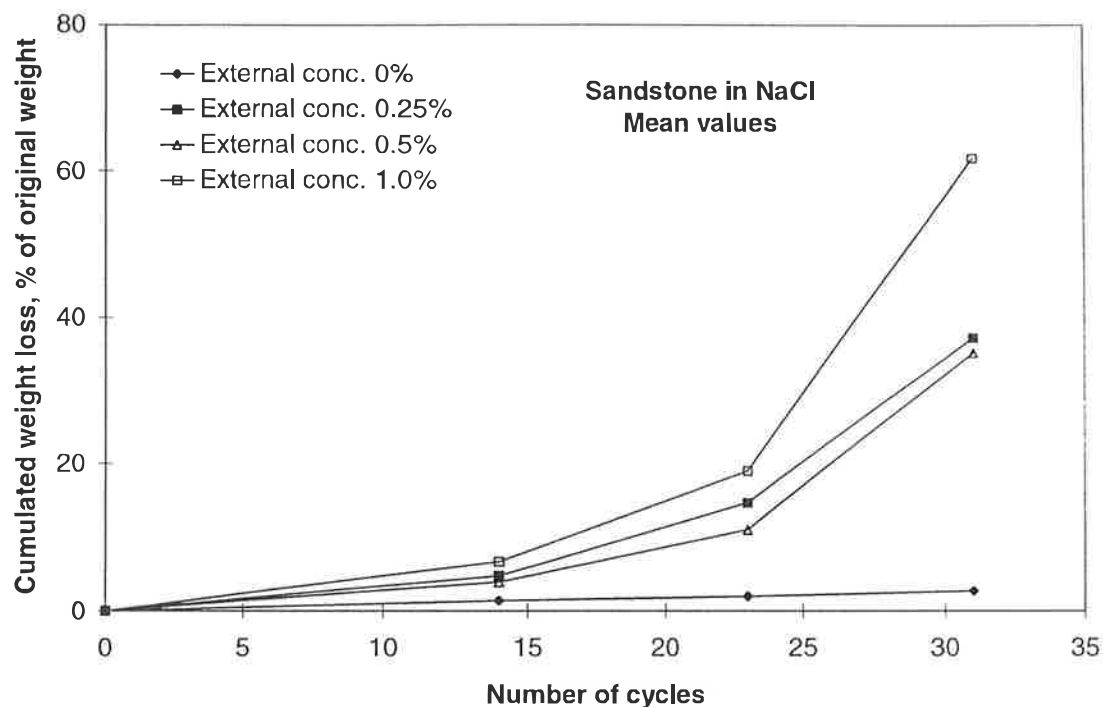


Fig. 4.1. Cumulated weight loss as function of number of freeze-thaw cycles of sandstone with mean values of internal NaCl-concentration and different external concentration of NaCl.

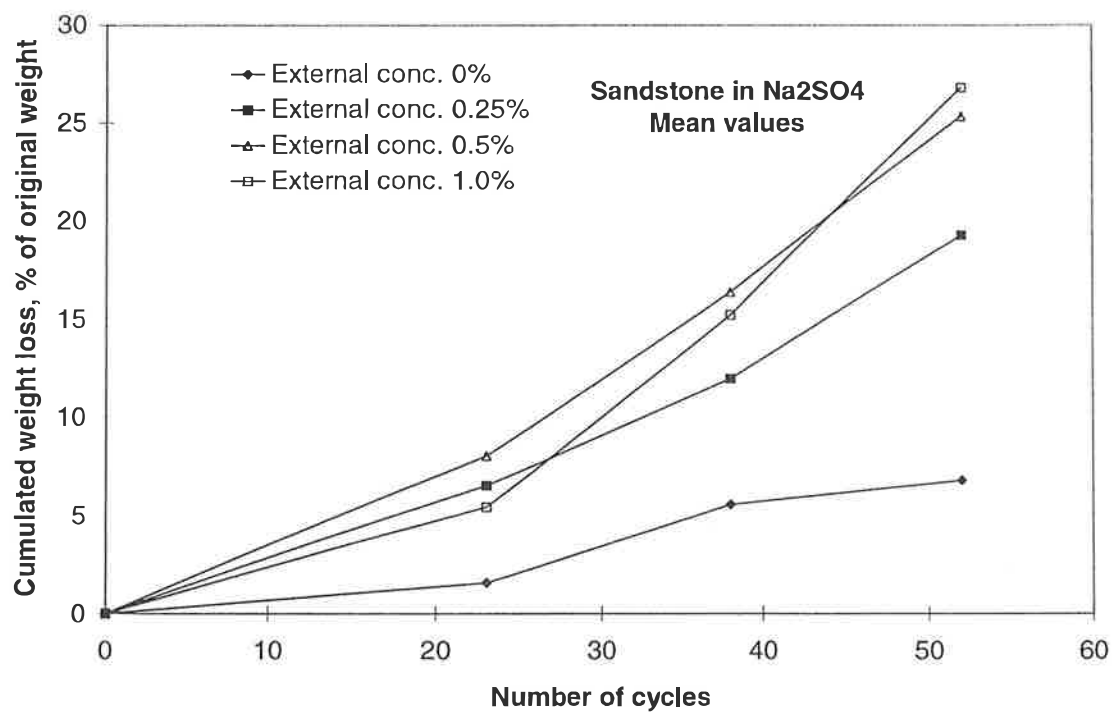


Fig. 4.2. Cumulated weight loss as function of number of freeze-thaw cycles of sandstone with mean values of internal Na<sub>2</sub>SO<sub>4</sub>-concentration and different external concentration of Na<sub>2</sub>SO<sub>4</sub>.

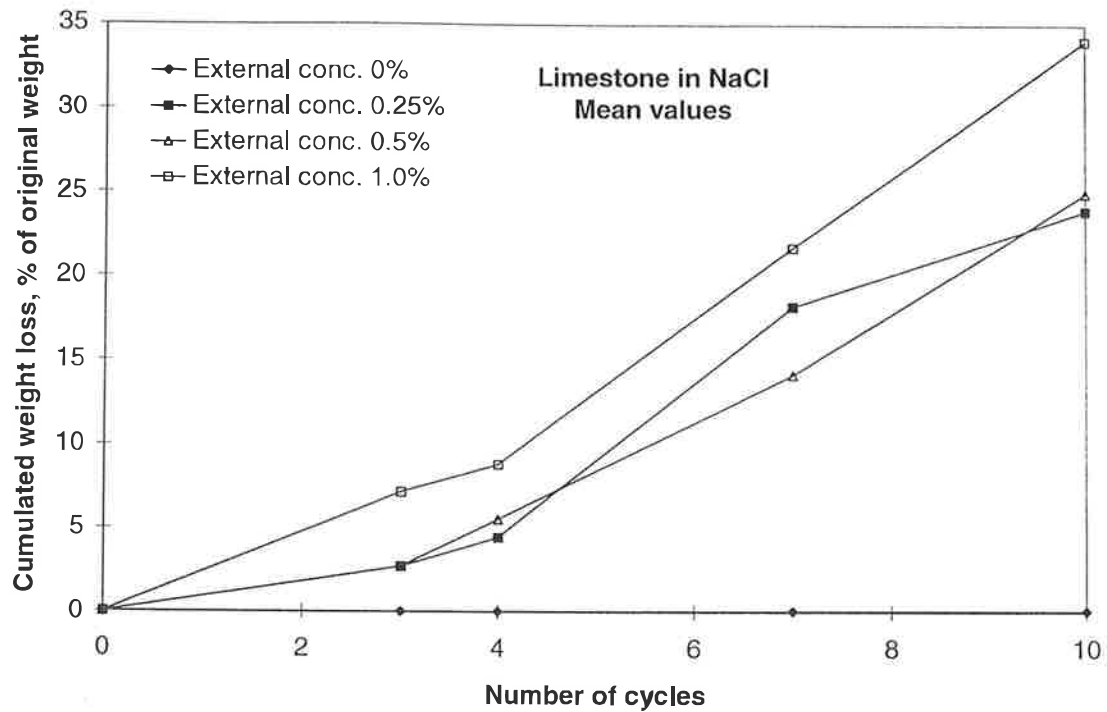


Fig. 4.3. Cumulated weight loss as function of number of freeze-thaw cycles of limestone with mean values of internal NaCl-concentration and different external concentration of NaCl.

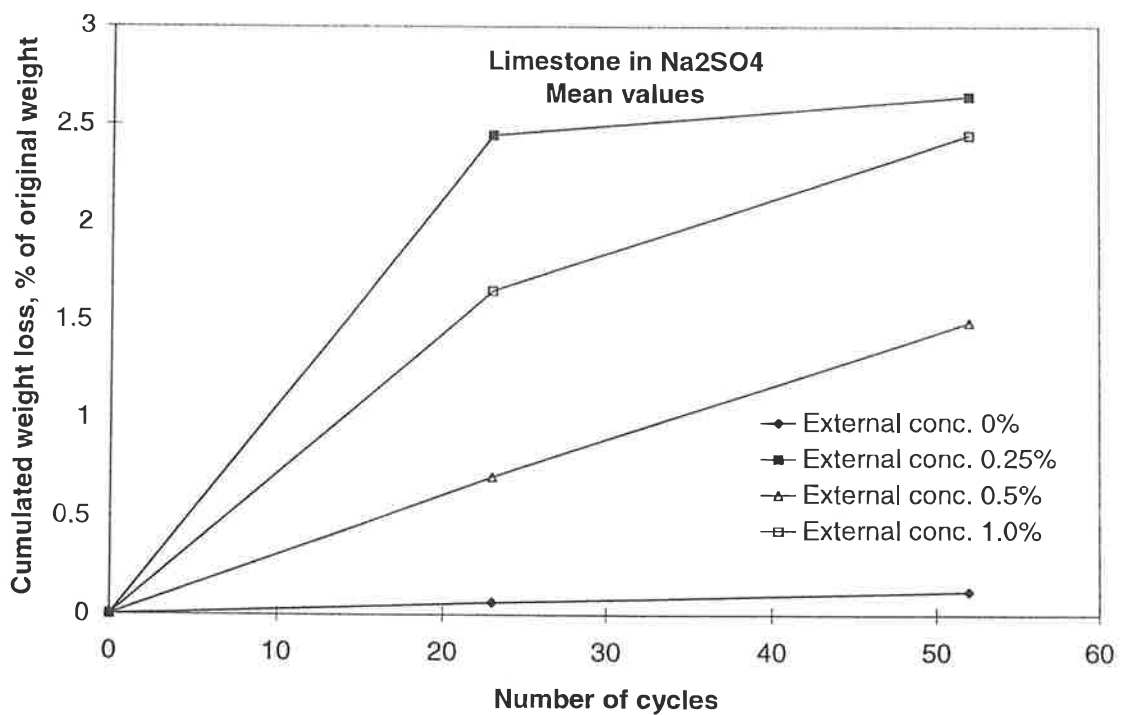


Fig. 4.4. Cumulated weight loss as function of number of freeze-thaw cycles of limestone with mean values of internal Na<sub>2</sub>SO<sub>4</sub>-concentration and different external concentration of Na<sub>2</sub>SO<sub>4</sub>.

*Weight loss (fig. 2.13-2.16 and 4.2)*

The internal concentration has no significant effect on the external damage. The mean values in fig. 4.2 show that the damage at external concentration  $0\% < 0.25\% < 0.5\%$ . (1% is insignificant.)

#### **4.3 Limestone in NaCl**

*Internal Damage (fig. 2.17-2.20)*

No internal damage could be detected. The resonant frequency was hard to measure, depending on changes of the dimensions of the prisms, caused by huge weight loss.

*Weight loss (fig. 2.21-2.24 and 4.3)*

A small tendency can be seen of more damage occurring when the internal solution is pure water. The mean values in fig. 4.3 show that the damage at external concentration  $0\% \ll 0.25\%$  and  $0.5\%$  (no significant difference between these two concentrations)  $< 1\%$ .

#### **4.4 Limestone in Na<sub>2</sub>SO<sub>4</sub>**

*Internal Damage (fig. 2.25-2.28)*

The internal damage of limestone in Na<sub>2</sub>SO<sub>4</sub> is detectable, but relatively small. Neither the internal nor the external concentration have effect on the internal damage.

*Weight loss (fig. 2.29-2.32 and 4.4)*

The external damage of limestone in Na<sub>2</sub>SO<sub>4</sub> is detectable, but relatively small. There is no significant difference between internal concentrations. Few, large pieces fall off. The mean values in fig. 4.4 show that the damage at external concentration  $0\% < 0.5\% < 1\% < 0.25\%$ , but as the weight loss is very small, this is almost insignificant.

#### **4.5 Comments**

Damage (both internal and external) increase with increasing number of freeze-thaw cycles. The increase is normally linear. There is no correlation between internal and external damage.

External concentrations have considerably more effect on weight loss than internal. External pure water give almost no damage compared to external salt solution. There is a very small difference between the concentrations 0.25%, 0.5% and 1.0%.

Internal concentration has normally no visible effect on scaling. The only exception from this rule is sandstone in pure water, where the internal concentration of NaCl has visible effect.

Neither the internal nor the external concentration normally have effect on the internal damage. The only exception from this rule is sandstone in pure water, which has less internal damage than has sandstone surrounded by NaCl-solution.

When there is a sudden "break" in the graph it depends on a broken sample – a large piece has fallen off or the prism has broken into two pieces.

## 5. References

1. Wessman, L. *Deterioration of Natural Stone by Freezing and Thawing in Salt Solutions*, Proceedings from the 7th International Congress on Durability of Building Materials and Components, Vol. 1, Stockholm 1996.
2. Lindmark, S. (1993) *Inverkan på testresultatet av variationer i saltkoncentrationer, saltfördelningar och fryscykelutformning vid saltfrostprovning enligt SS 13 72 44* (*Influence on test result of variations in salt concentration, salt distribution and freeze-thaw cycle at freeze-thaw test according to Swedish Standard SS 13 72 44*), Report TVBM - 7055, Division of Building Materials, University of Lund, Lund Institute of Technology.
3. Wessman, L. and Carlsson, T. (1995) *Karakterisering av några svenska naturstenar med tunnslipsmikroskopi* (*Characterisation of some Swedish Natural Stones with Thin Section Microscopy*), Report TVBM - 7095, Division of Building Materials, University of Lund, Lund Institute of Technology.
4. Nord, A. G. and Tronner, K. (1991) The Central Board of National Antiquities and the National Historical Museums, Conservation Institute, Report RIK 4, *Stone Weathering, Air pollution effects evidenced by chemical analysis*.



## Appendix 2

Loss in dynamic modulus of elasticity  
and cumulated weight loss  
versus number of freeze-thaw cycles

Note the different scales on the axes.

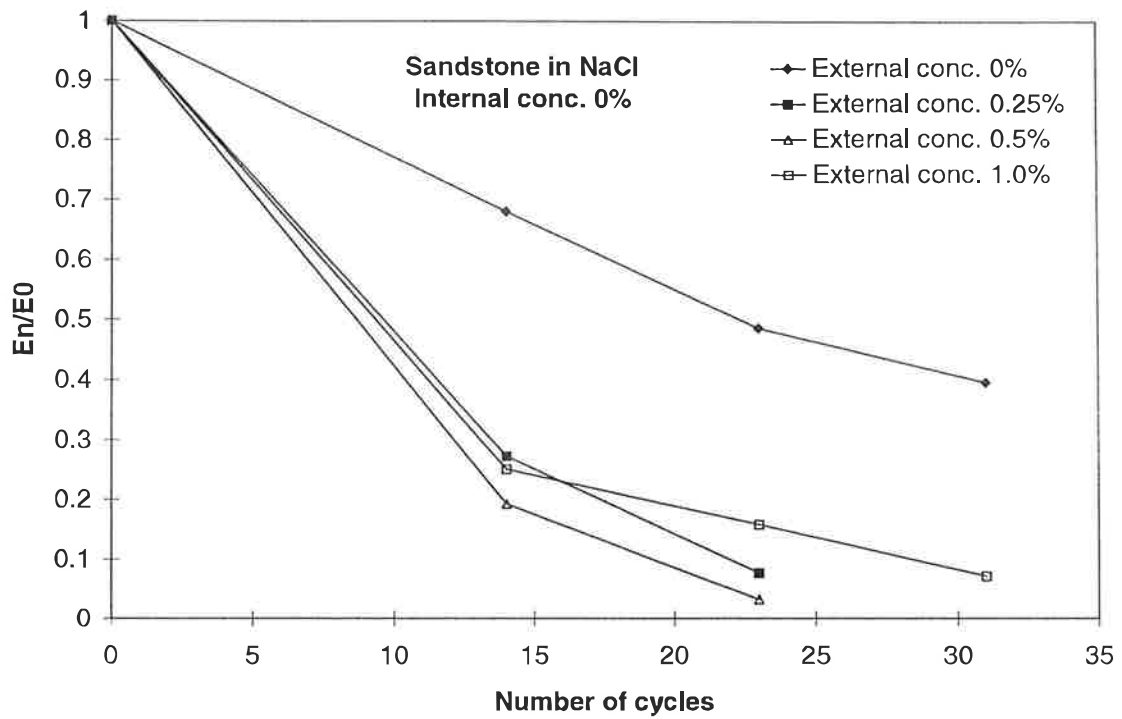


Fig 2.1. Loss in dynamic modulus of elasticity versus number of freeze-thaw cycles of sandstone with different external NaCl-concentration and internal concentration of 0% NaCl.

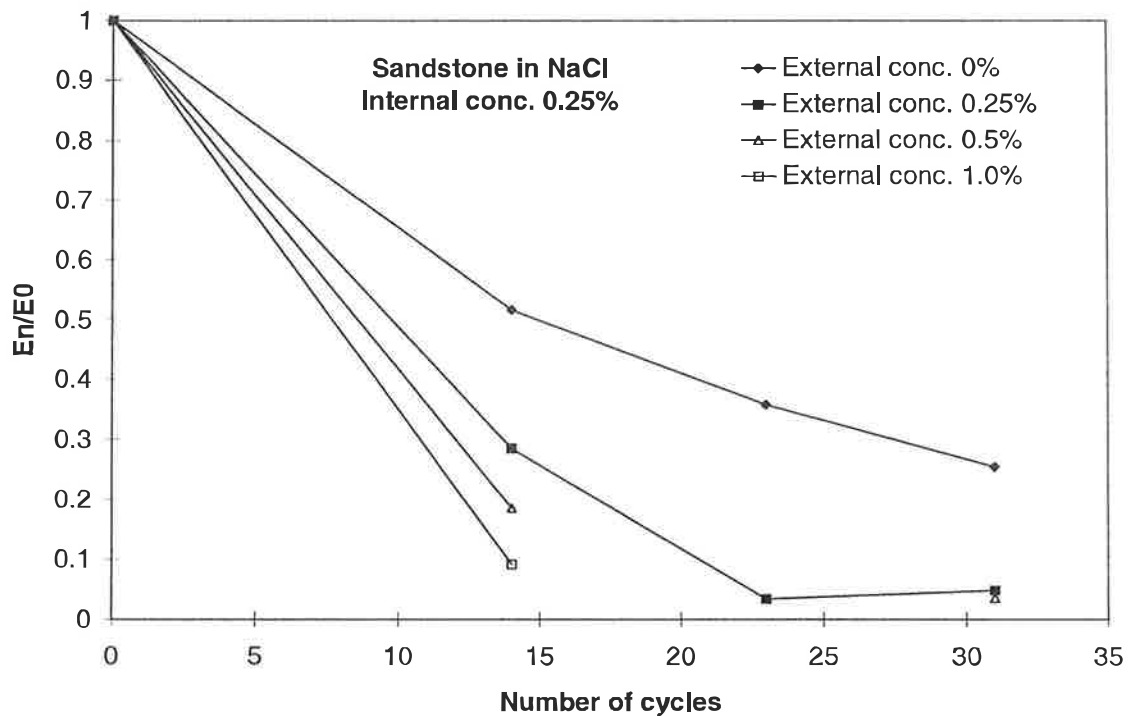


Fig 2.2. Loss in dynamic modulus of elasticity versus number of freeze-thaw cycles of sandstone with different external NaCl-concentration and internal concentration of 0.25% NaCl.

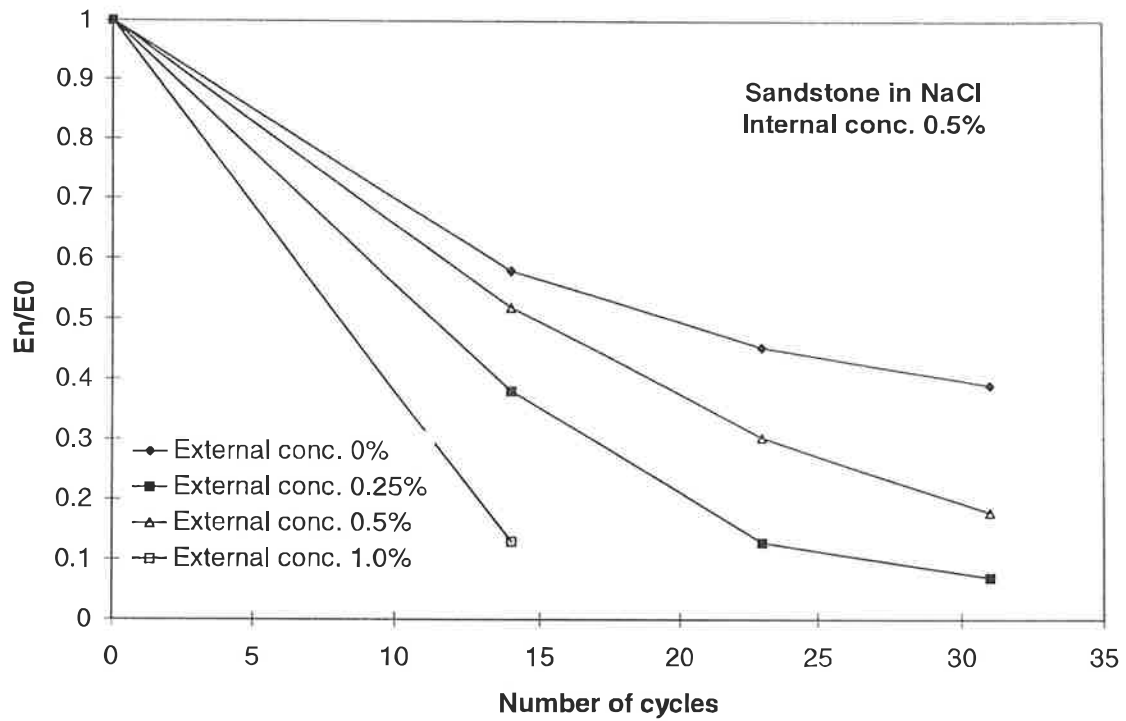


Fig 2.3. Loss in dynamic modulus of elasticity versus number of freeze-thaw cycles of sandstone with different external NaCl-concentration and internal concentration of 0.5% NaCl.

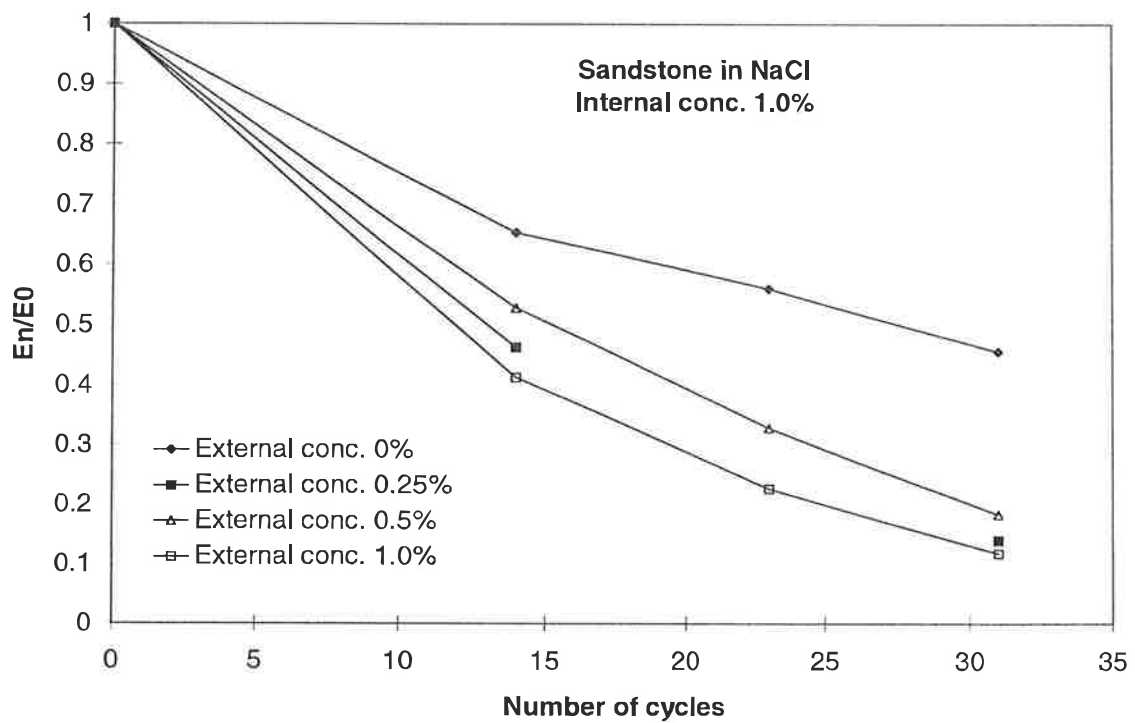


Fig 2.4. Loss in dynamic modulus of elasticity versus number of freeze-thaw cycles of sandstone with different external NaCl-concentration and internal concentration of 1.0% NaCl.

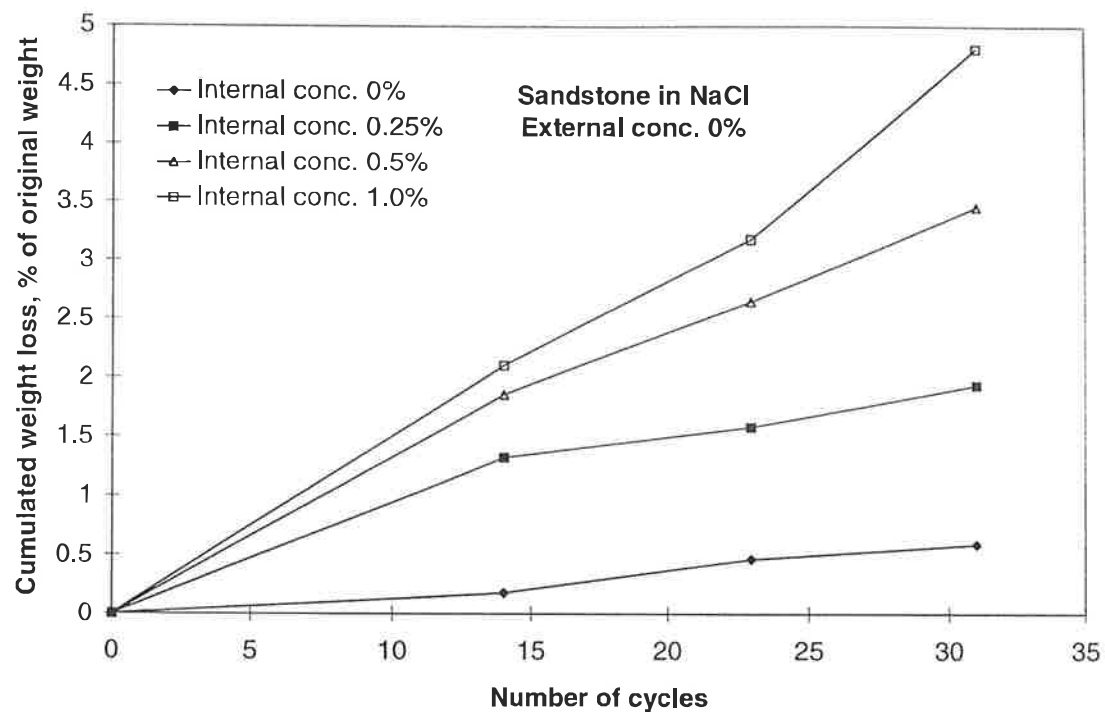


Fig 2.5. Cumulated weight loss versus number of freeze-thaw cycles of sandstone with different internal NaCl-concentration and external concentration of 0% NaCl.

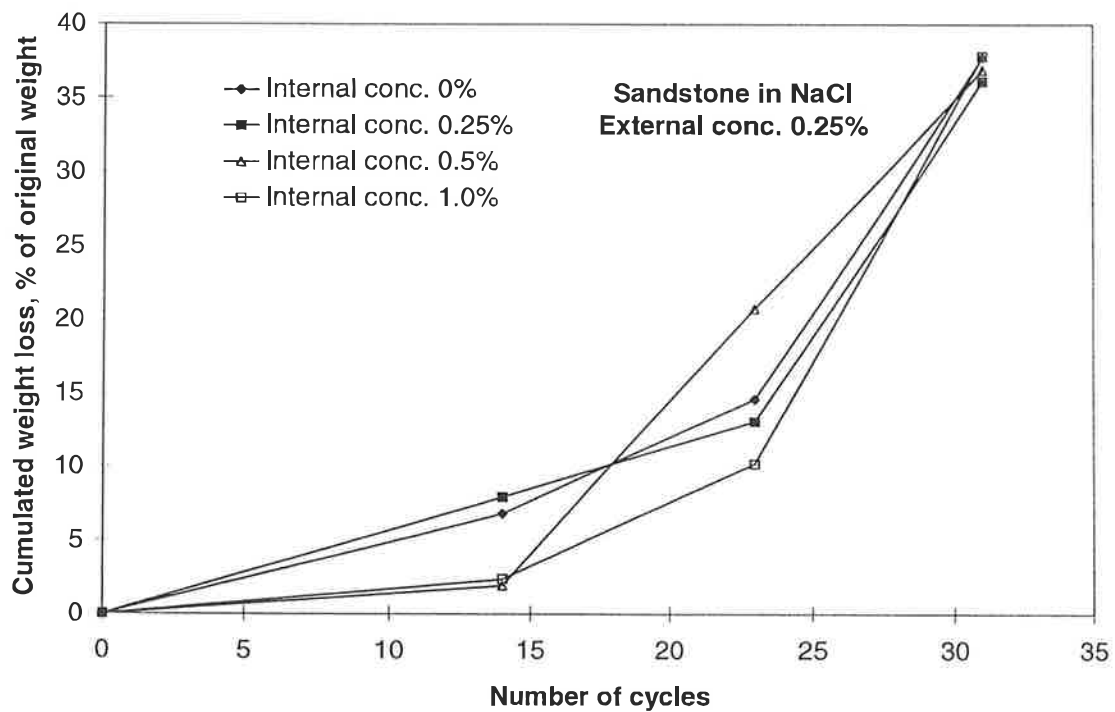


Fig 2.6. Cumulated weight loss versus number of freeze-thaw cycles of sandstone with different internal NaCl-concentration and external concentration of 0.25% NaCl.

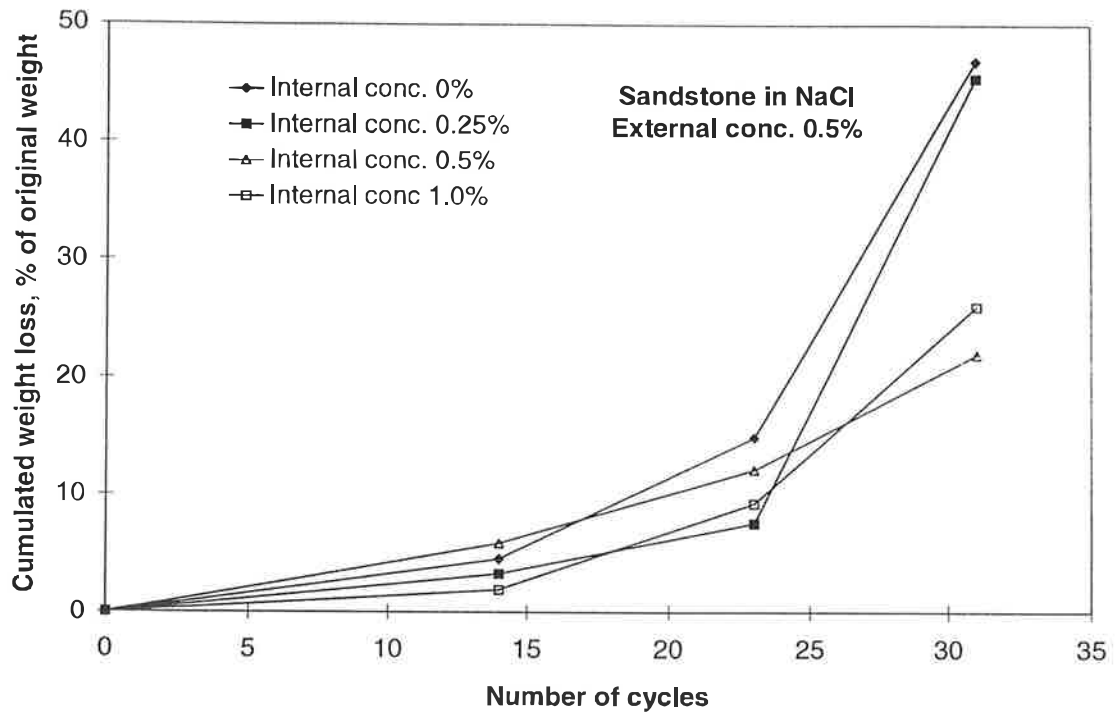


Fig 2.7. Cumulated weight loss versus number of freeze-thaw cycles of sandstone with different internal NaCl-concentration and external concentration of 0.5% NaCl.

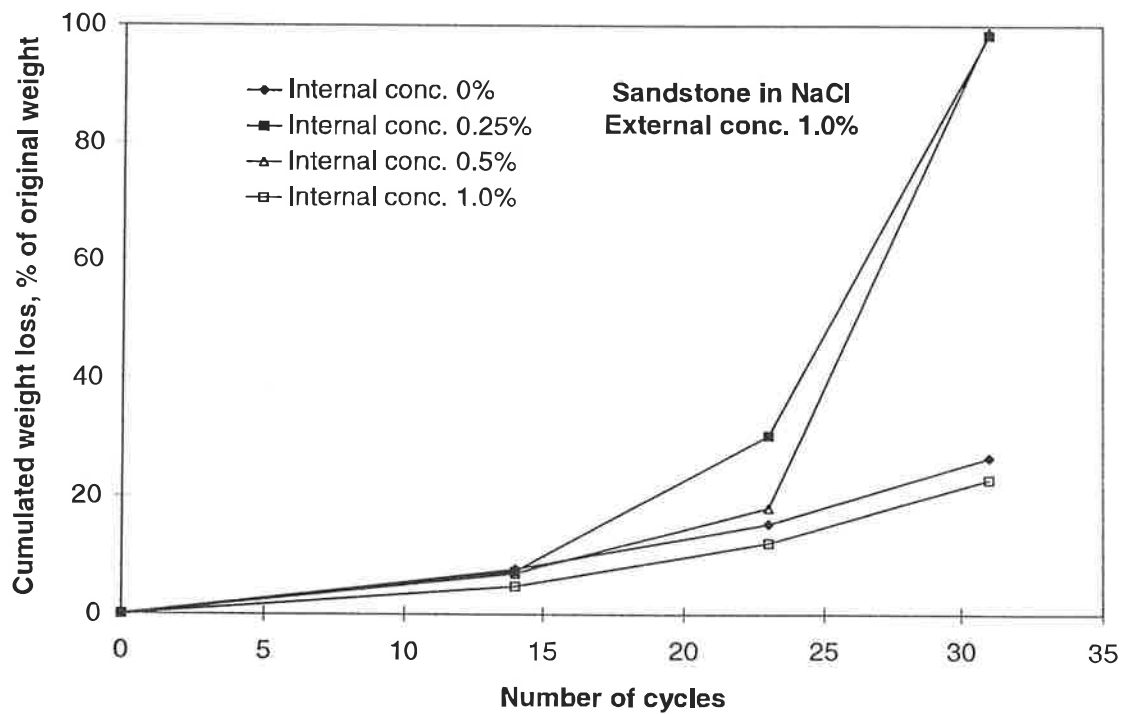


Fig 2.8. Cumulated weight loss versus number of freeze-thaw cycles of sandstone with different internal NaCl-concentration and external concentration of 1.0% NaCl.

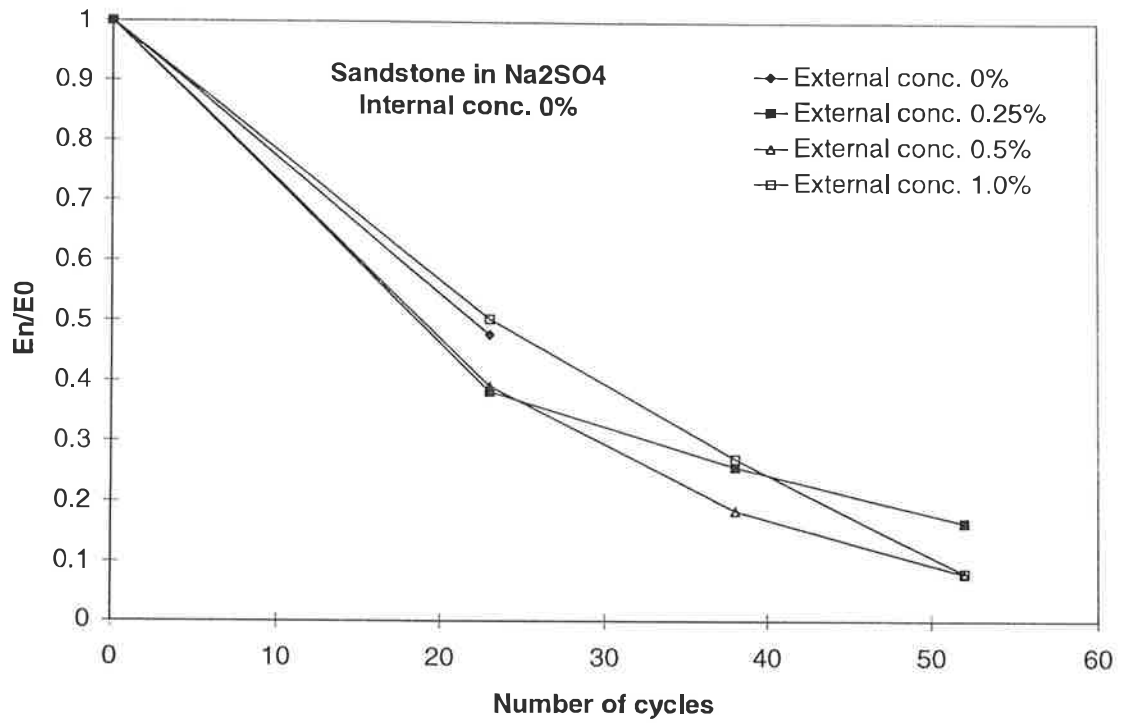


Fig 2.9. Loss in dynamic modulus of elasticity versus number of freeze-thaw cycles of sandstone with different external Na<sub>2</sub>SO<sub>4</sub>--concentration and internal concentration of 0% Na<sub>2</sub>SO<sub>4</sub>.

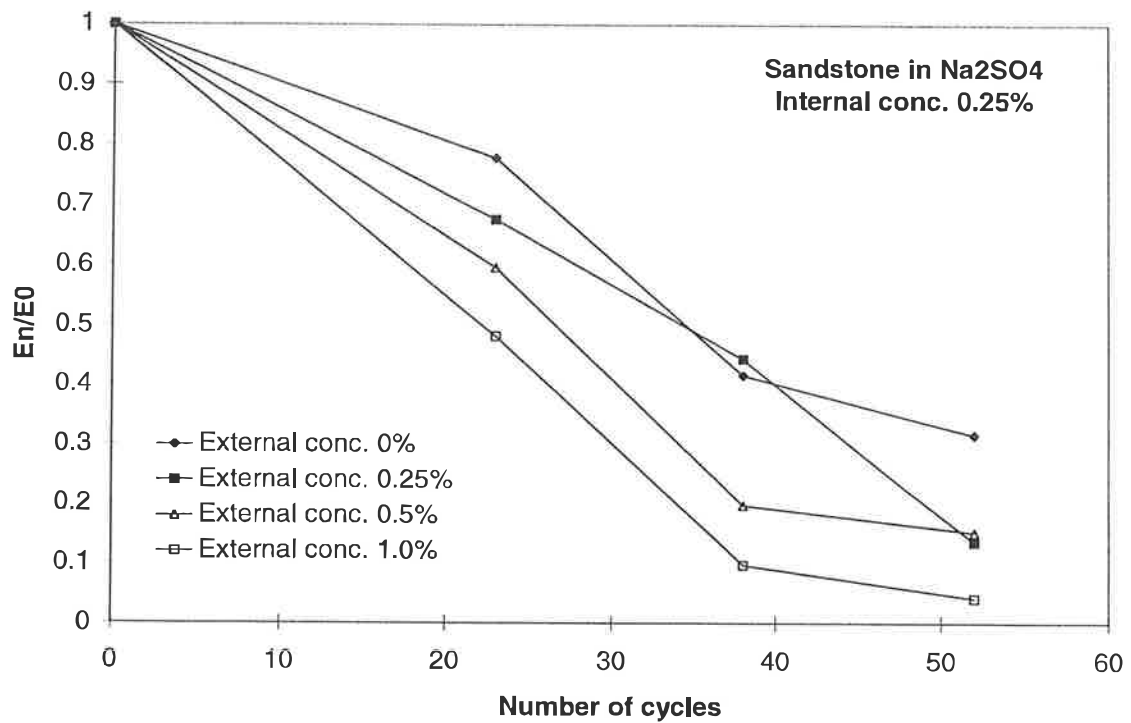


Fig 2.10. Loss in dynamic modulus of elasticity versus number of freeze-thaw cycles of sandstone with different external Na<sub>2</sub>SO<sub>4</sub>--concentration and internal concentration of 0.25% Na<sub>2</sub>SO<sub>4</sub>.

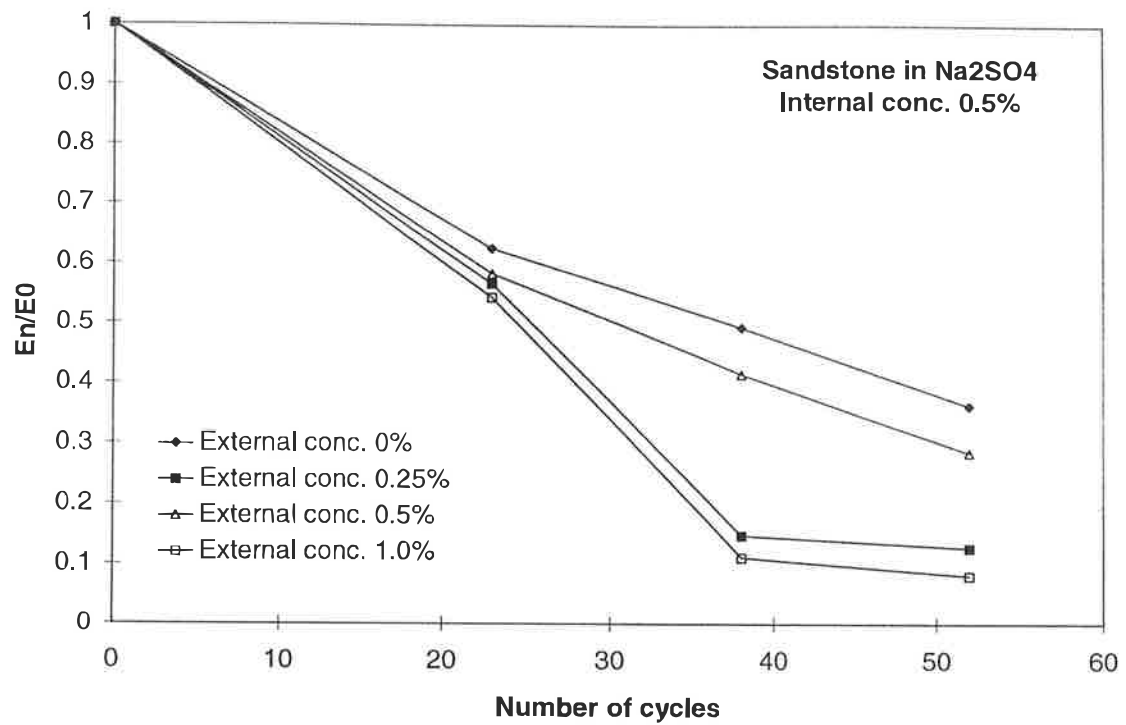


Fig 2.11. Loss in dynamic modulus of elasticity versus number of freeze-thaw cycles of sandstone with different external  $\text{Na}_2\text{SO}_4$ --concentration and internal concentration of 0.5%  $\text{Na}_2\text{SO}_4$ .

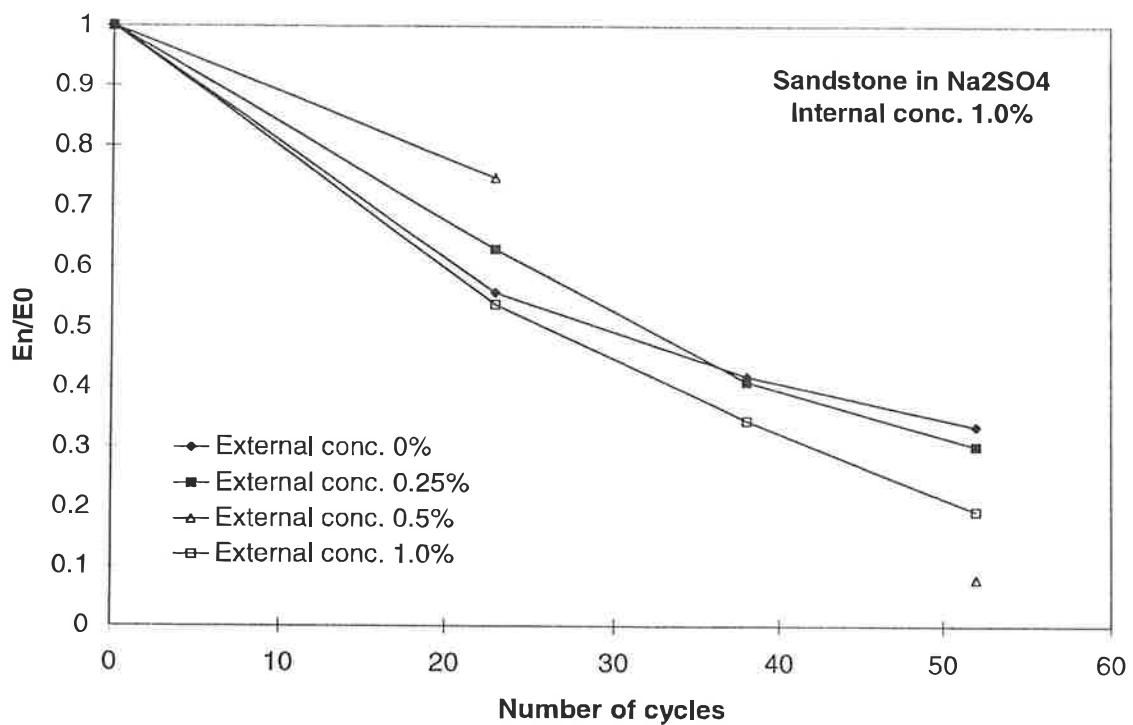


Fig 2.12. Loss in dynamic modulus of elasticity versus number of freeze-thaw cycles of sandstone with different external  $\text{Na}_2\text{SO}_4$ --concentration and internal concentration of 1.0%  $\text{Na}_2\text{SO}_4$ .

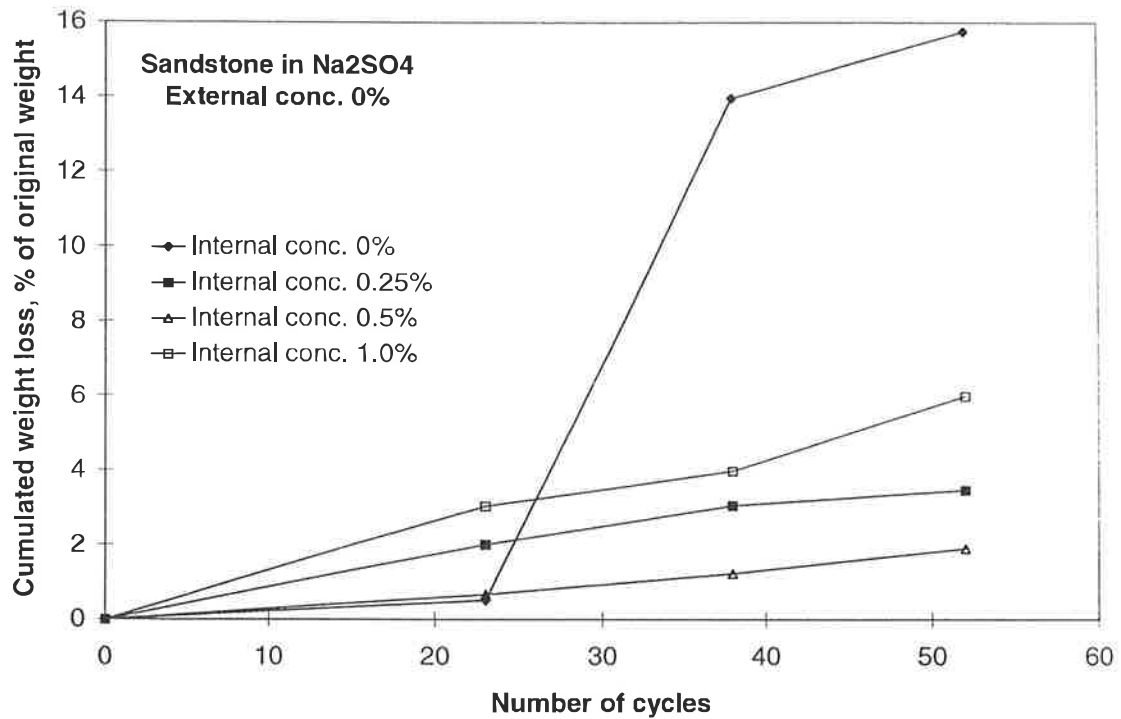


Fig 2.13. Cumulated weight loss versus number of freeze-thaw cycles of sandstone with different internal Na<sub>2</sub>SO<sub>4</sub>-concentration and external concentration of 0% Na<sub>2</sub>SO<sub>4</sub>.

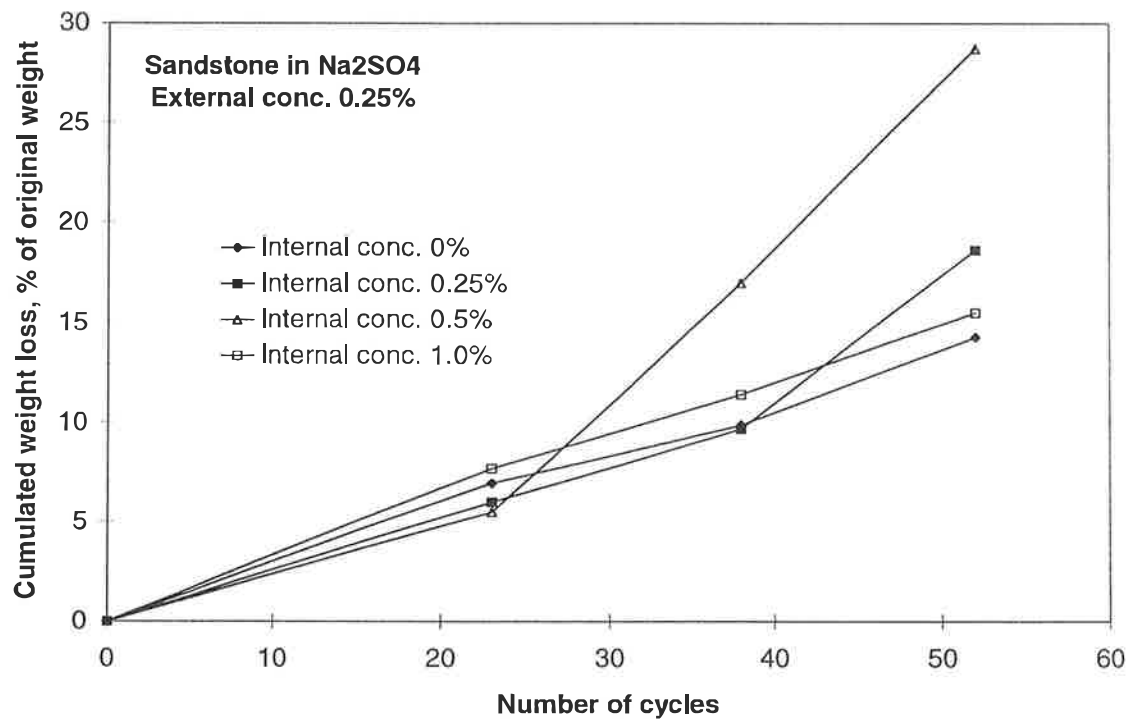


Fig 2.14. Cumulated weight loss versus number of freeze-thaw cycles of sandstone with different internal Na<sub>2</sub>SO<sub>4</sub>-concentration and external concentration of 0.25% Na<sub>2</sub>SO<sub>4</sub>.



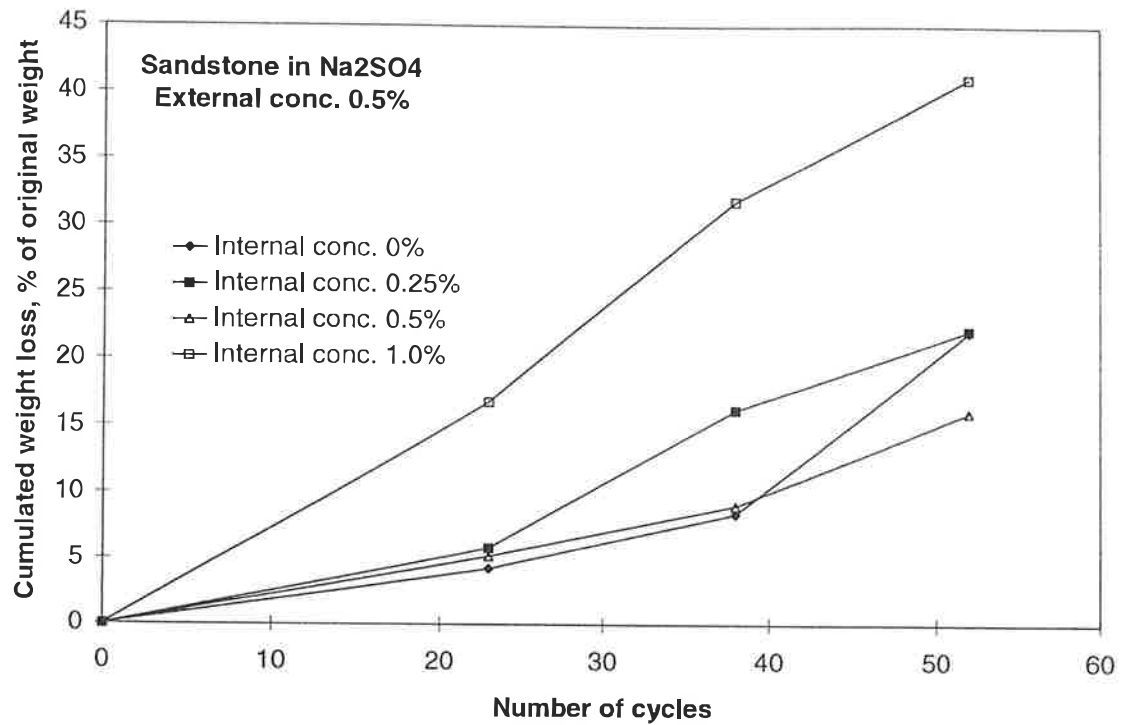


Fig 2.15. Cumulated weight loss versus number of freeze-thaw cycles of sandstone with different internal Na<sub>2</sub>SO<sub>4</sub>-concentration and external concentration of 0.5% Na<sub>2</sub>SO<sub>4</sub>.

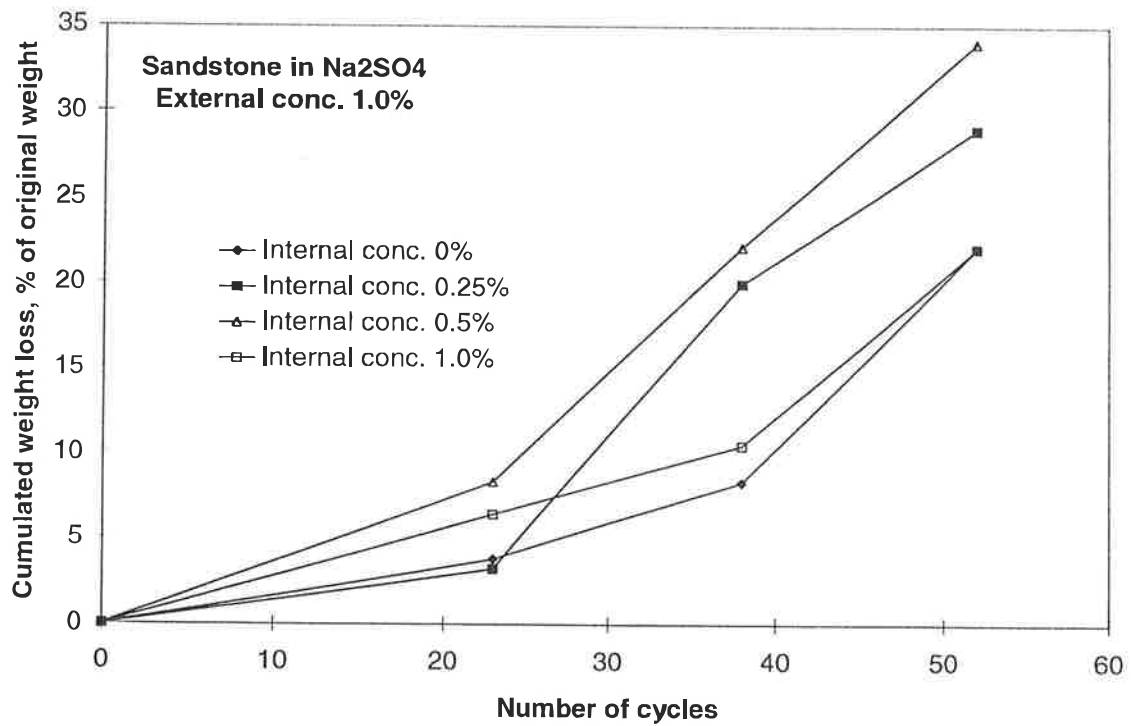


Fig 2.16. Cumulated weight loss versus number of freeze-thaw cycles of sandstone with different internal Na<sub>2</sub>SO<sub>4</sub>-concentration and external concentration of 1.0% Na<sub>2</sub>SO<sub>4</sub>.

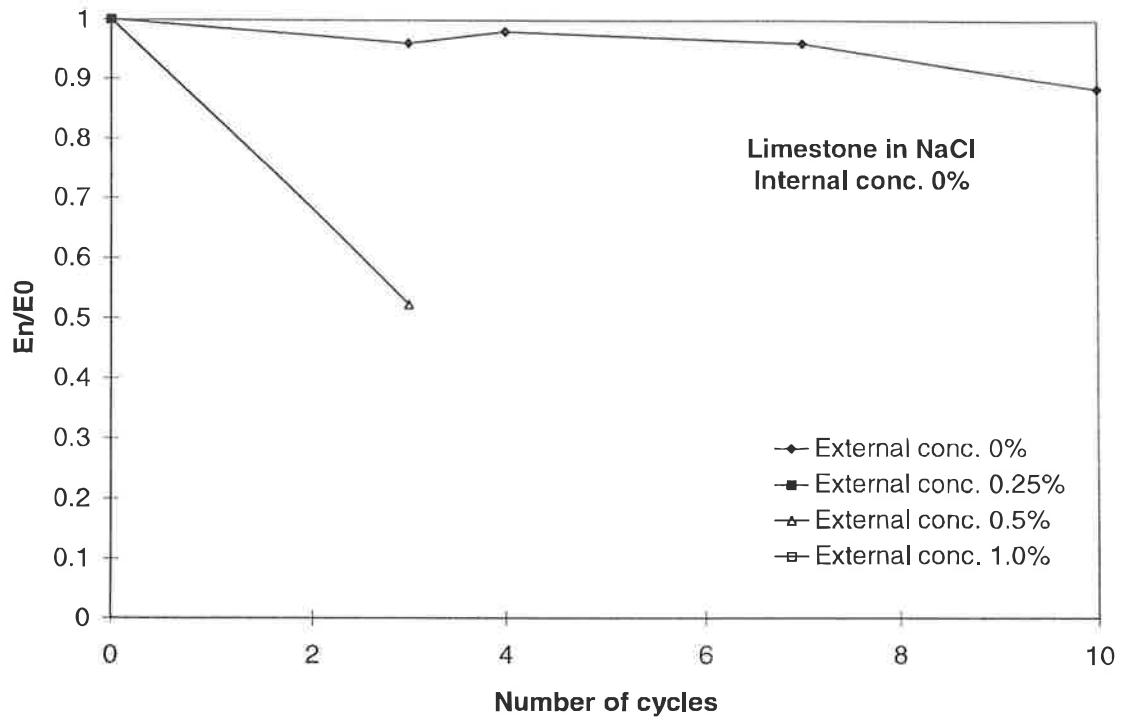


Fig 2.17. Loss in dynamic modulus of elasticity versus number of freeze-thaw cycles of limestone with different external NaCl-concentration and internal concentration of 0% NaCl.

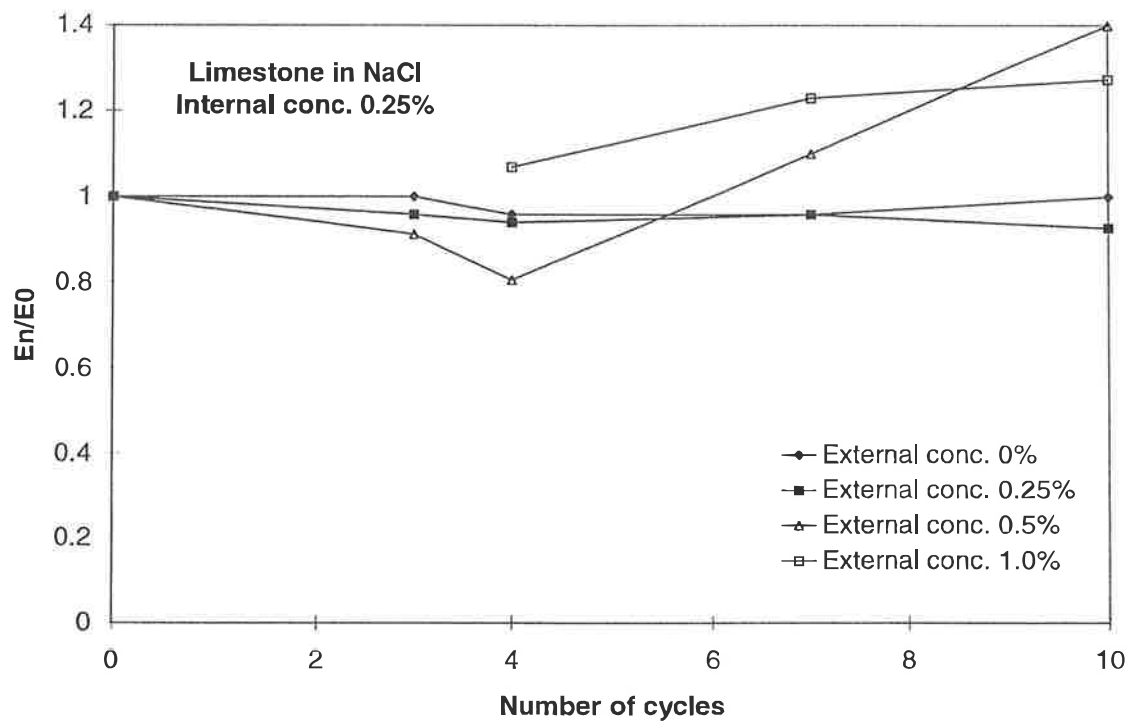


Fig 2.18. Loss in dynamic modulus of elasticity versus number of freeze-thaw cycles of limestone with different external NaCl-concentration and internal concentration of 0.25% NaCl.

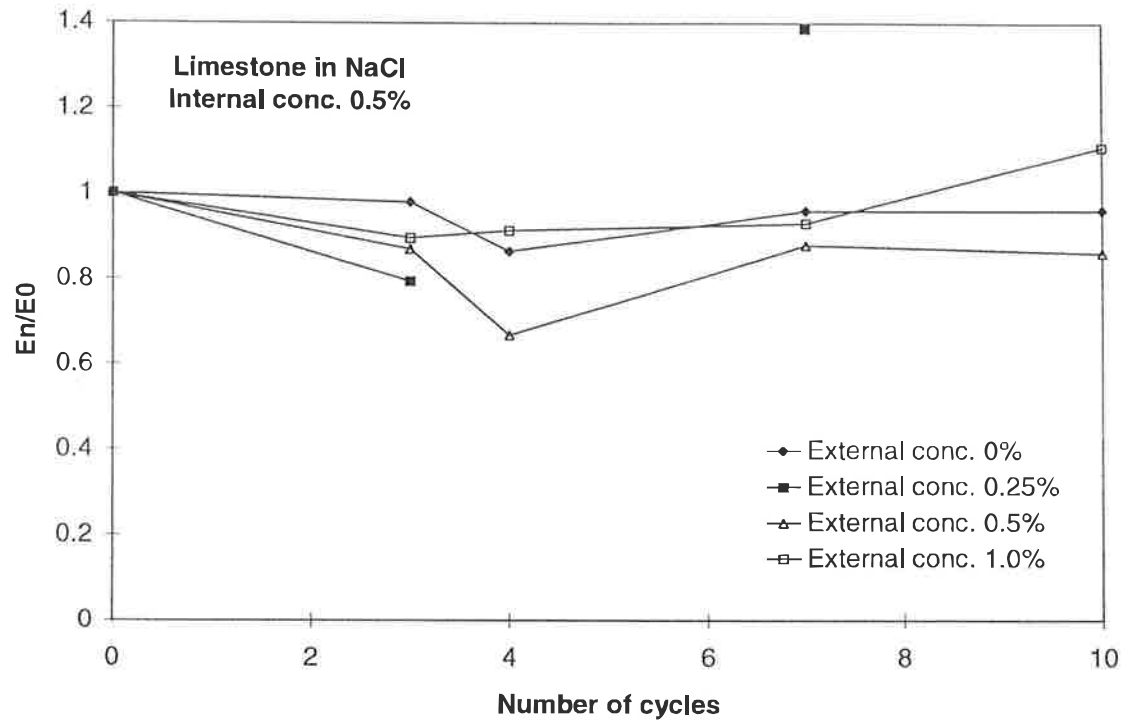


Fig 2.19. Loss in dynamic modulus of elasticity versus number of freeze-thaw cycles of limestone with different external NaCl-concentration and internal concentration of 0.5% NaCl.

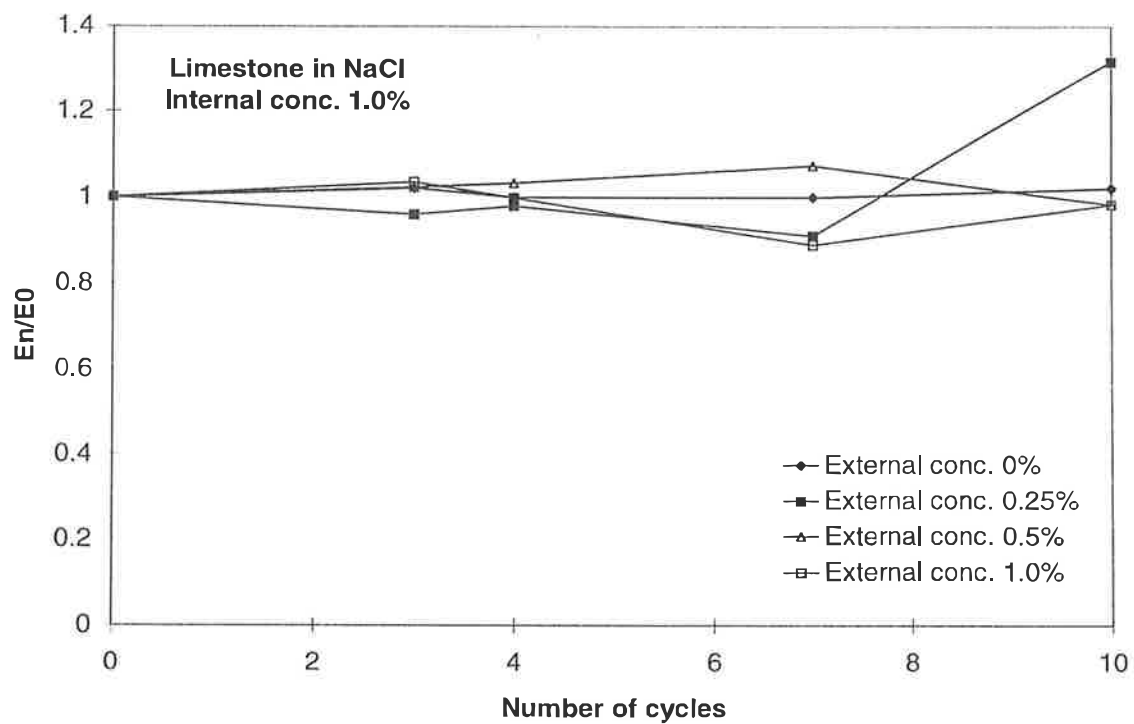


Fig 2.20. Loss in dynamic modulus of elasticity versus number of freeze-thaw cycles of limestone with different external NaCl-concentration and internal concentration of 1.0% NaCl.

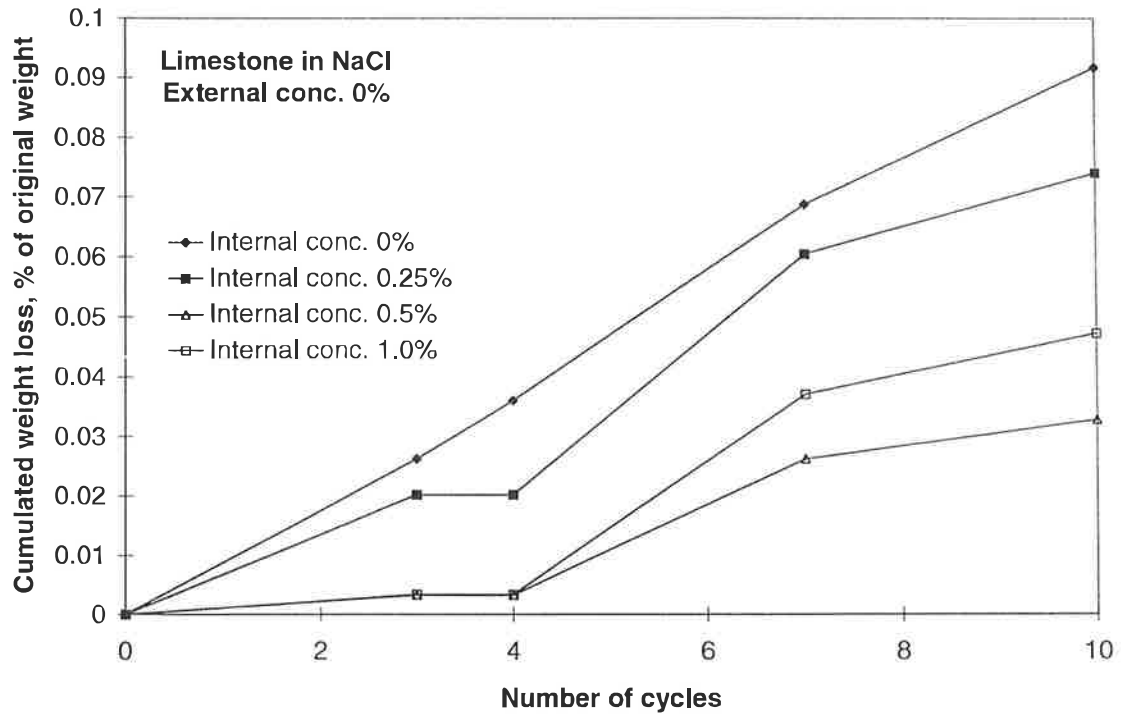


Fig 2.21. Cumulated weight loss versus number of freeze-thaw cycles of limestone with different internal NaCl-concentration and external concentration of 0% NaCl.

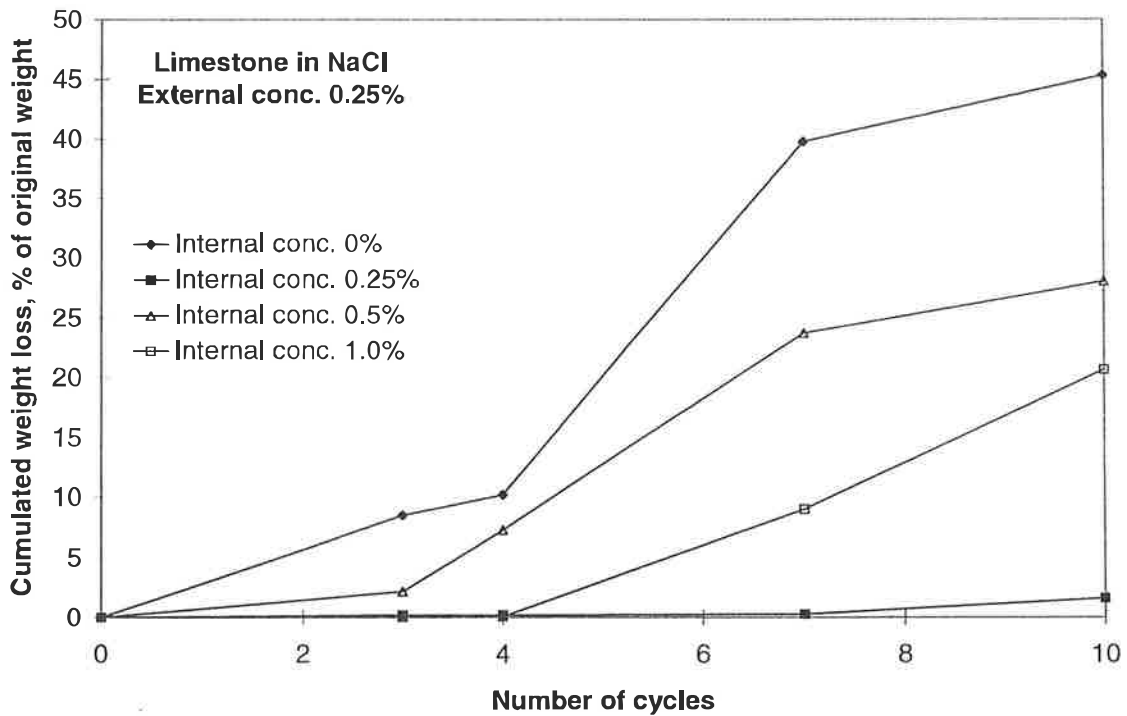


Fig 2.22. Cumulated weight loss versus number of freeze-thaw cycles of limestone with different internal NaCl-concentration and external concentration of 0.25% NaCl.

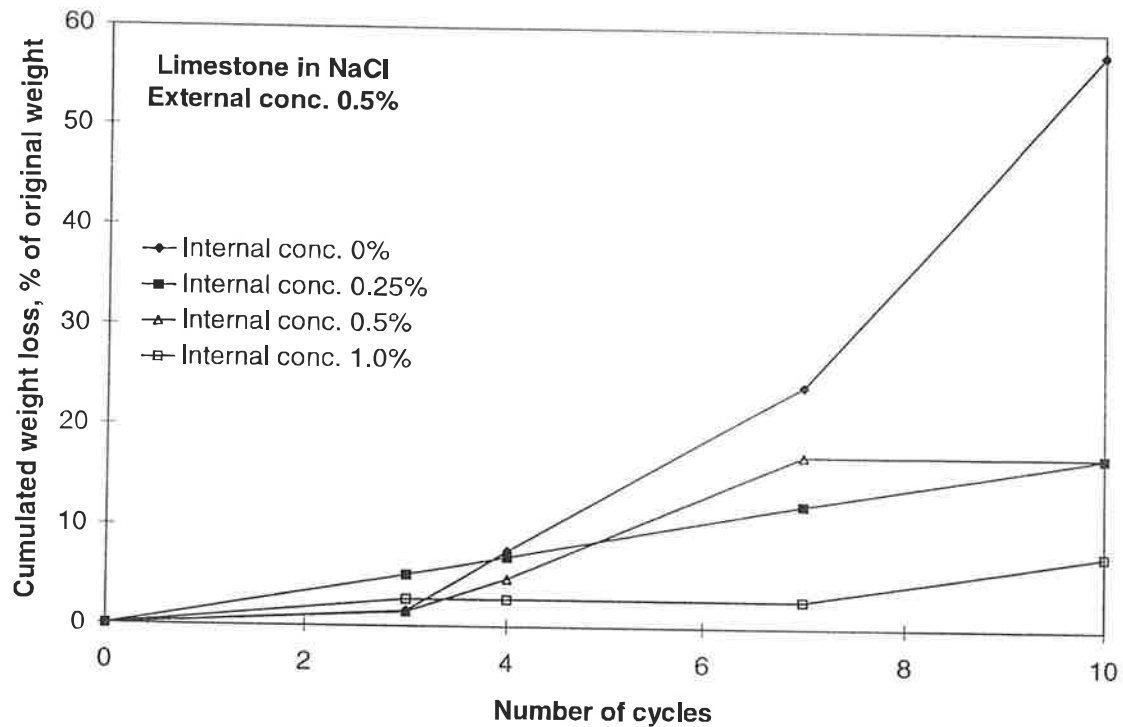


Fig 2.23. Cumulated weight loss versus number of freeze-thaw cycles of limestone with different internal NaCl-concentration and external concentration of 0.5% NaCl.

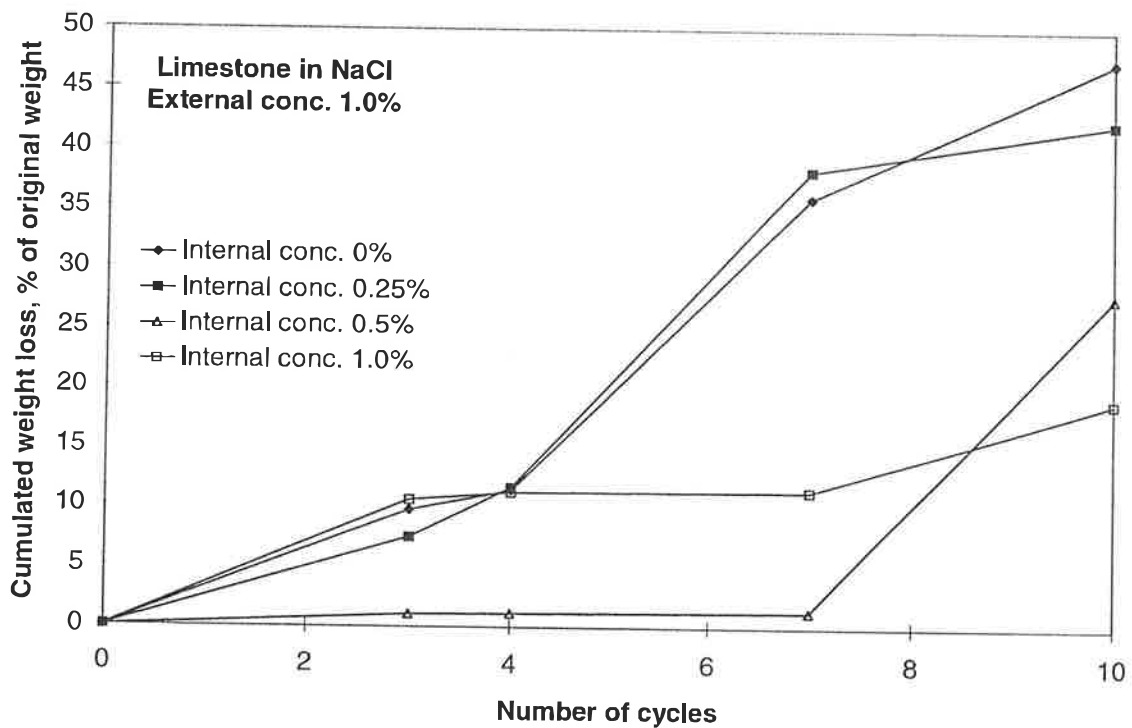


Fig 2.24. Cumulated weight loss versus number of freeze-thaw cycles of limestone with different internal NaCl-concentration and external concentration of 1.0% NaCl.

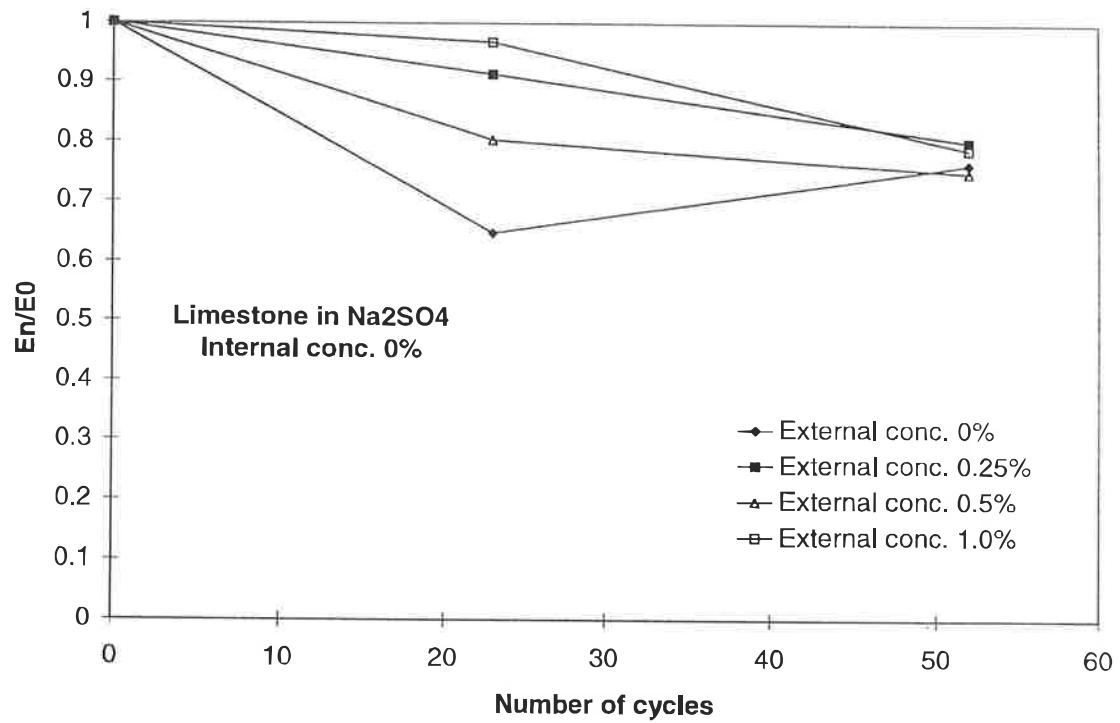


Fig 2.25. Loss in dynamic modulus of elasticity versus number of freeze-thaw cycles of limestone with different external  $\text{Na}_2\text{SO}_4$ -concentration and internal concentration of 0%  $\text{Na}_2\text{SO}_4$ .

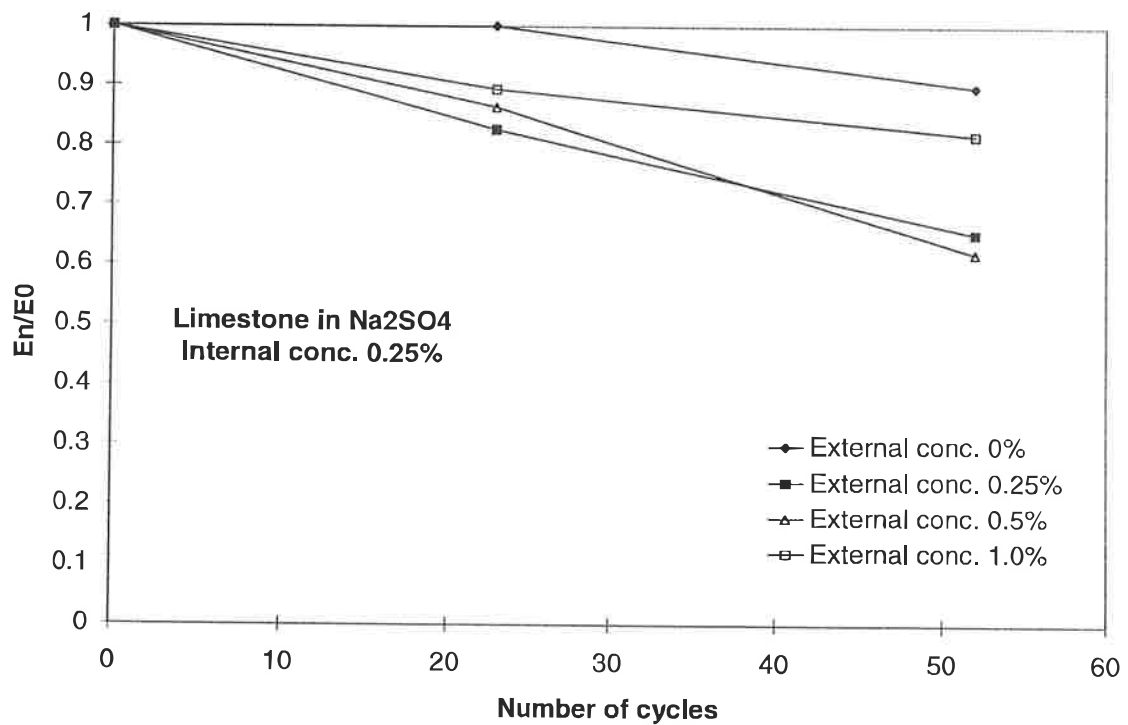


Fig 2.26. Loss in dynamic modulus of elasticity versus number of freeze-thaw cycles of limestone with different external  $\text{Na}_2\text{SO}_4$ -concentration and internal concentration of 0.25%  $\text{Na}_2\text{SO}_4$ .

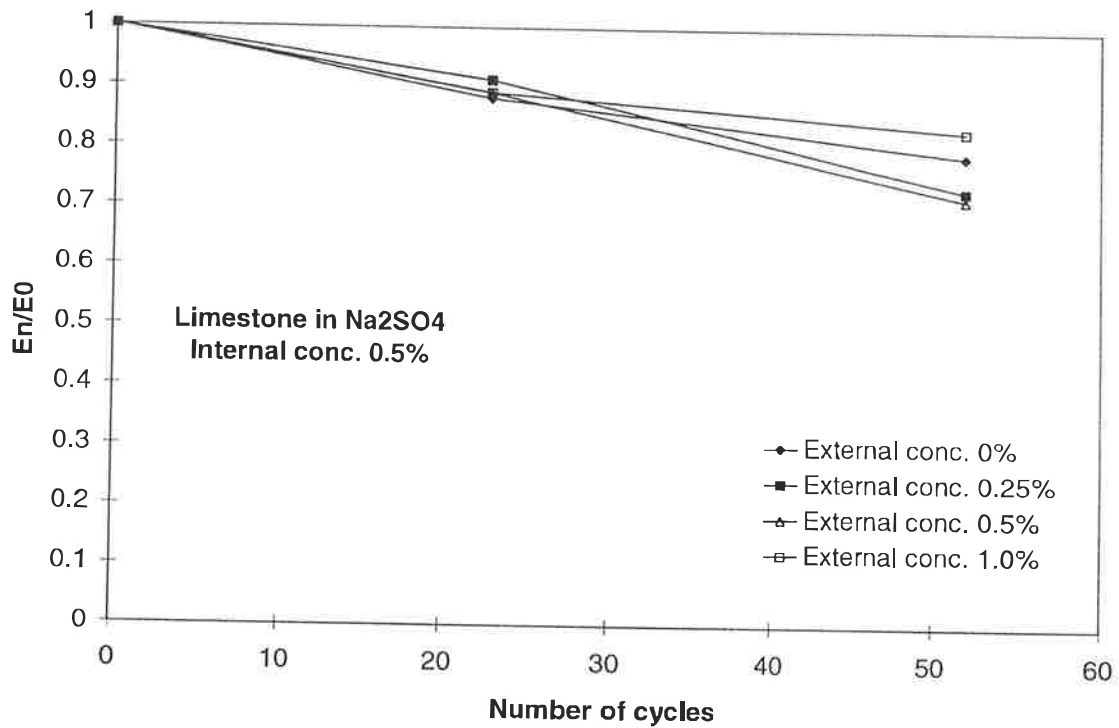


Fig 2.27. Loss in dynamic modulus of elasticity versus number of freeze-thaw cycles of limestone with different external  $\text{Na}_2\text{SO}_4$ -concentration and internal concentration of 0.5%  $\text{Na}_2\text{SO}_4$ .

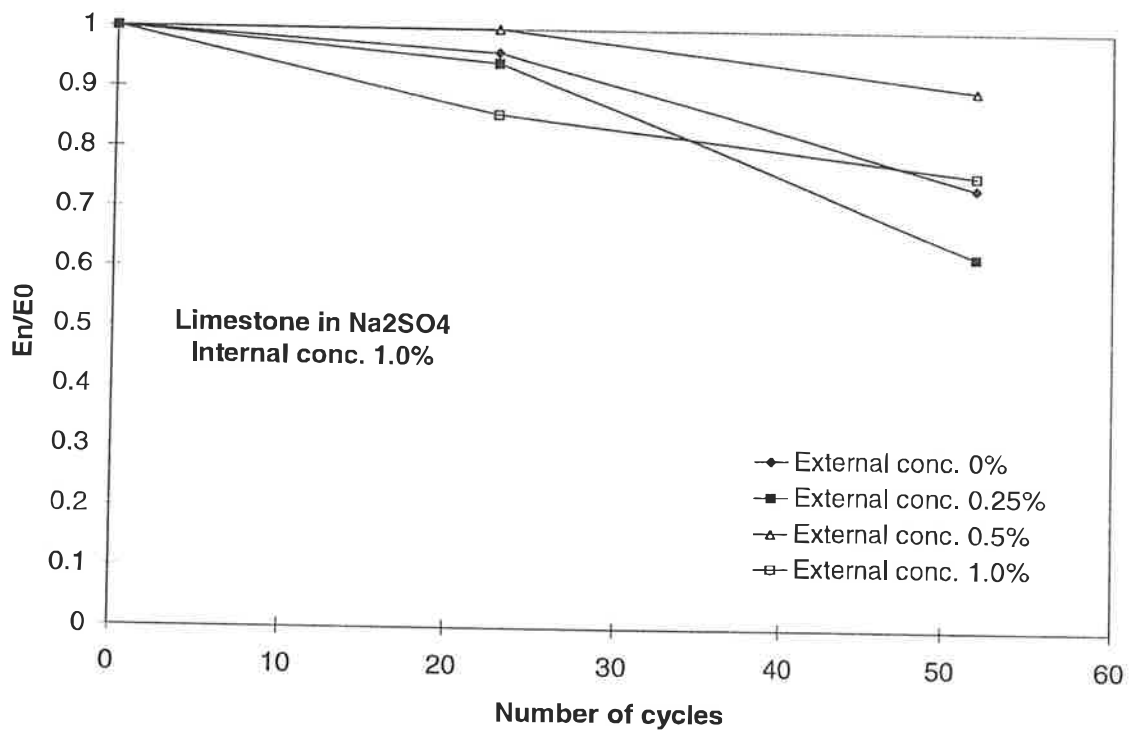


Fig 2.28. Loss in dynamic modulus of elasticity versus number of freeze-thaw cycles of limestone with different external  $\text{Na}_2\text{SO}_4$ -concentration and internal concentration of 1.0%  $\text{Na}_2\text{SO}_4$ .

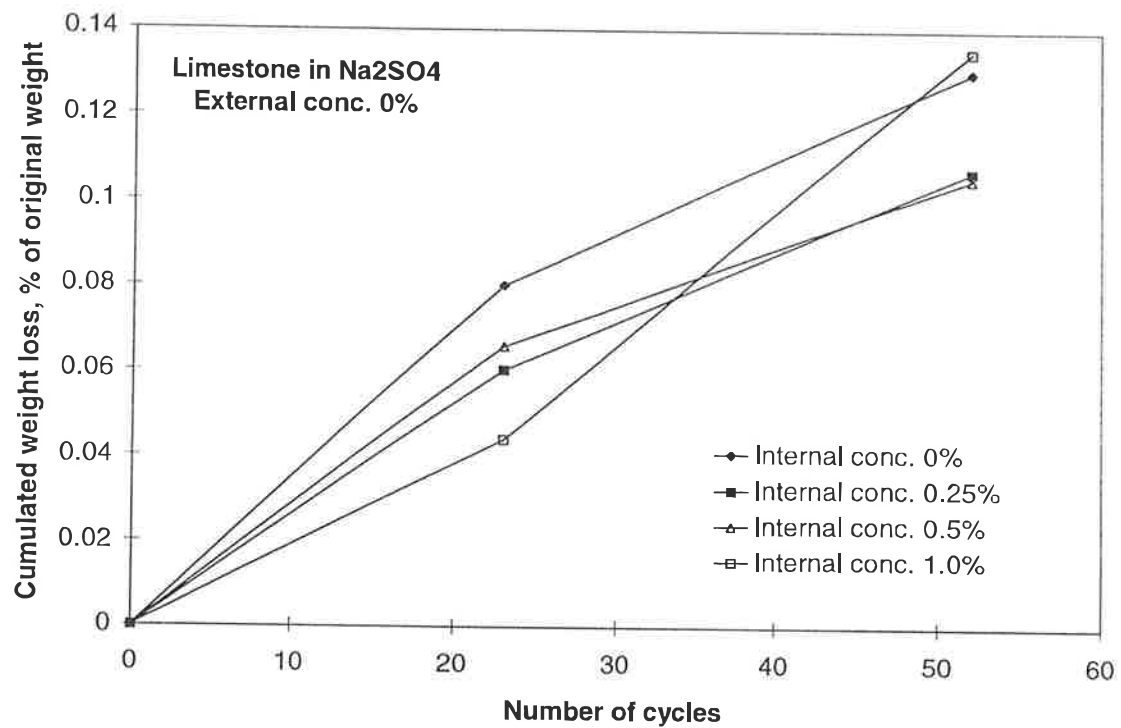


Fig 2.29. Cumulated weight loss versus number of freeze-thaw cycles of limestone with different internal Na<sub>2</sub>SO<sub>4</sub>-concentration and external concentration of 0% Na<sub>2</sub>SO<sub>4</sub>.

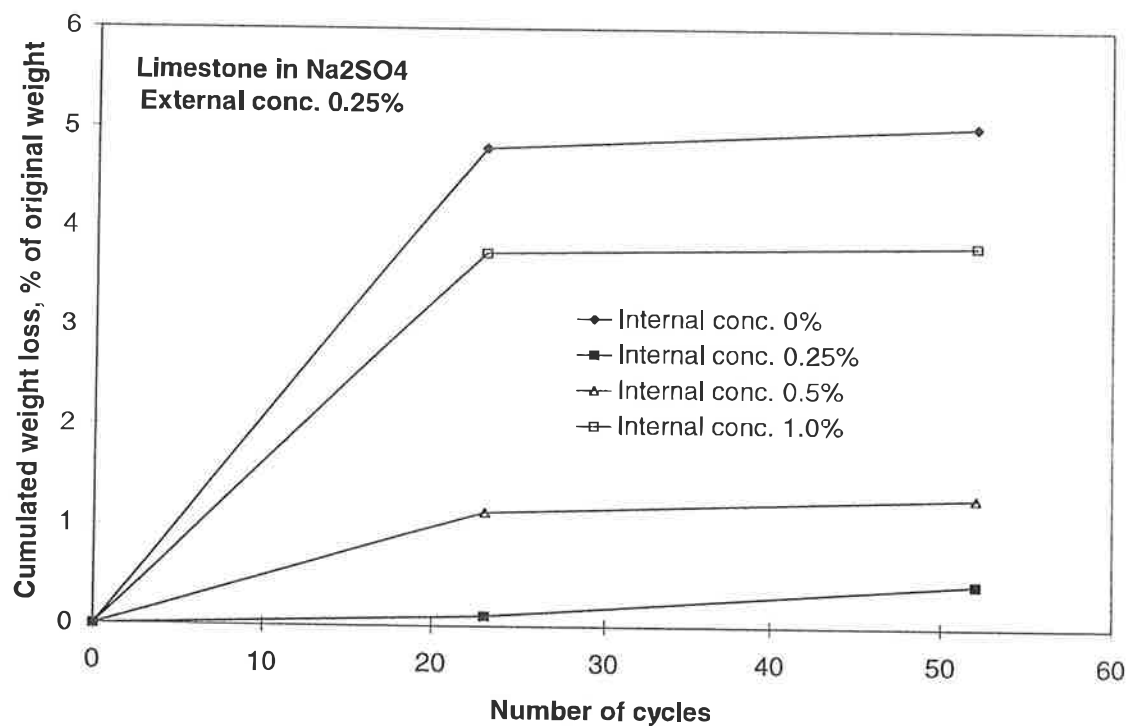


Fig 2.30. Cumulated weight loss versus number of freeze-thaw cycles of limestone with different internal Na<sub>2</sub>SO<sub>4</sub>-concentration and external concentration of 0.25% Na<sub>2</sub>SO<sub>4</sub>.



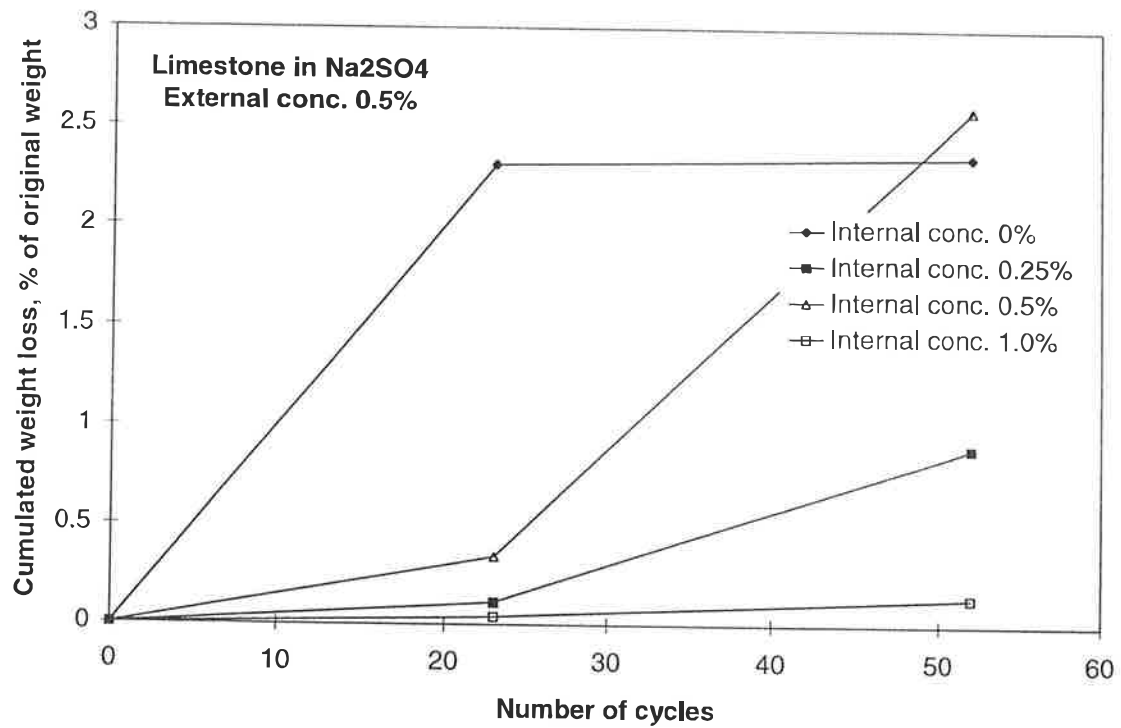


Fig 2.31. Cumulated weight loss versus number of freeze-thaw cycles of limestone with different internal Na<sub>2</sub>SO<sub>4</sub>-concentration and external concentration of 0.5% Na<sub>2</sub>SO<sub>4</sub>.

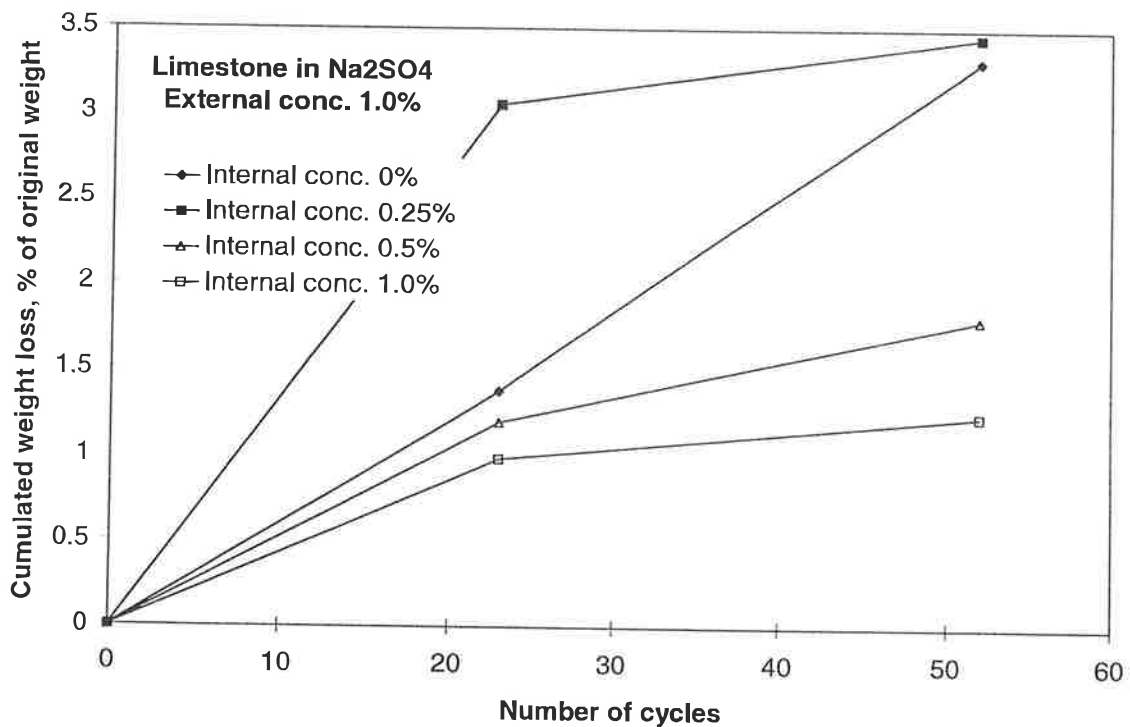


Fig 2.32. Cumulated weight loss versus number of freeze-thaw cycles of limestone with different internal Na<sub>2</sub>SO<sub>4</sub>-concentration and external concentration of 1.0% Na<sub>2</sub>SO<sub>4</sub>.